

NASA 100-111P  
11-75  
233324  
NASA CASE NO. MFS-28368-1  
PRINT FIGURE 1

NOTICE

108.

The invention disclosed in this document resulted from research in aeronautical and space activities performed under programs of the National Aeronautics and Space Administration. The invention is owned by NASA and is, therefore, available for licensing in accordance with the NASA Patent Licensing Regulation (14 Code of Federal Regulations 1245.2).

To encourage commercial utilization of NASA-owned inventions, it is NASA policy to grant licenses to commercial concerns. Although NASA encourages nonexclusive licensing to promote competition and achieve the widest possible utilization, NASA will consider the granting of a limited exclusive license, pursuant to the NASA Patent Licensing Regulations, when such a license will provide the necessary incentive to the licensee to achieve early practical application of the invention.

Address inquiries and all applications for license for this invention to NASA Patent Counsel, Marshall Space Flight Center, Mail Code CCO1, Huntsville, AL 35812. Approved NASA forms for application for nonexclusive or exclusive license are available from the above address.

MSFC

(NASA-Case-MFS-28368-1) METHOD AND  
APPARATUS FOR MAINTAINING THERMAL CONTROL IN  
PLASMA CONDITIONS Patent Application (NASA)  
10 D CSCL 201

N90-10717

Unclas  
65/75 0233324

## METHOD AND APPARATUS FOR MAINTAINING THERMAL CONTROL IN PLASMA CONDITIONS

### ORIGIN OF THE INVENTION

The invention described herein was made by an  
5 employee of the United States Government and may be  
manufactured and used by or for the Government for  
governmental purposes without the payment of any royalties  
thereon or therefor.

### TECHNICAL FIELD

10 This application is generally directed to thermal  
control in plasma conditions and more particularly to an  
apparatus and method for determining the effects of  
exposure of oxygen plasma on a film polymer or thin metals  
whose bulk is maintained at a predetermined temperature.

### 15 BACKGROUND OF THE INVENTION

The present invention concerns an apparatus and  
method for determining the effects of exposure of ionized  
gases, such as oxygen plasma, on a specimen such as a film  
polymer. Testing is done in a chamber in an oxygen plasma  
20 environment. The test apparatus includes a platform for  
support of the specimen in the chamber and also includes  
temperature control devices and temperature sensing devices  
which are used to maintain a predetermined temperature on  
the couple during its exposure to the oxygen environment.

25 The use of test chambers for testing samples under  
various conditions are known in the art. A hyperbolic  
simulator, for example, is disclosed in U.S. Patent No.  
3,817,109 which is used to evaluate thickness change,  
stretch-flex change, thermal conductance and other material  
30 properties under simulated deep sea pressures down to 1,000  
feet. The chamber includes a thermal conductance tester.

Another type of test chamber is disclosed in U.S.  
Patent No. 3,545,242 which is directed to a flammability  
test chamber for testing materials in various

environments. The material is tested to determine the flame propagation of the material. Tests are also performed to determine in certain mechanical movements of the apparatus will create a spark in the material.

5 Patent No. 4,425,810 discloses a chamber in which physical properties of a sample material is measured at controllable temperatures and pressure. The chamber includes a sample holder in combination with chamber heating and cooling means.

10 None of the above discussed patents disclose applicant's structure relating generally to the concept of a sample holder for use in a chamber having a plasma therein. Specifically, none of the patents are directed to, as is applicant's method and apparatus, to such a  
15 chamber having a closure member which serves as a support for a specimen supporting structure which also supports temperature sensing and temperature controlling means. Also, none of the patents are directed to a method and apparatus for determining the effects of exposure of oxygen  
20 plasma on a film polymer whose bulk is maintained at a predetermined temperature.

It is an object of the present invention, therefore, to provide an apparatus and method for determining the effects of a plasma on a specimen supported in a chamber.

25 It is a further object of the present invention to provide such apparatus and method for making such determination on a film polymer whose bulk is maintained at a predetermined temperature.

It is yet another object of the present invention to  
30 provide the chamber with temperature sensing and temperature controlling devices.

It is yet another object of the present invention to provide an oxygen plasma environment in the chamber for testing the specimen therein.

### BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is an elevational view, partially broken away, of the test apparatus of the present invention.

Figure 2 is a plan view, partially broken away, of the test chamber of the present invention including the specimen and support structure therefor.

### DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring now to the drawings, there is shown a reaction chamber 8 in which oxygen plasma is produced. The reaction chamber 8 carries a specimen 10, which may be a film polymer. The wall 12 of the chamber is typically made of Pyrex glass or the like. The chamber is connected to a source of RF energy 14 that provides up to 100 watts of continuous 13.56 MHz power into the chamber. The RF energy is used to dissociate molecular oxygen supplied into the chamber into oxygen plasma. A vacuum pump 16 is connected with the chamber. The reaction chamber is part of a small commercial reactor that with the exception of the external vacuum pump is a fully self-contained device sold under the trade name PLASMOD by the Tegal Corporation.

The test apparatus of the present invention includes a closure member 18 such as a plexiglass disc which serves as a support structure for the specimen 10 and for the thermal control apparatus used in the chamber. Closure member 18 is a hermetically sealed member which may be transparent so that conditions inside the chamber may be observed during the test procedure. The closure member serves as a door to the entrance opening 20 of the chamber and is adapted to fit within the entrance opening of the reaction chamber 8. A silicon seal 22 is used to seal the opening. Closure member 18 is silicon coated and supports the test apparatus 24 within the chamber.

The test apparatus 24 includes a cooling coil 26, typically copper, which is supported in closure member 18

and extends into chamber 8 to provide coolant to the test apparatus. A platform 28 is provided for support of specimen 10 and a glass plate protective cover 32 for the specimen. Cooling coil 26 includes portions 34 and 36 to  
5 which platform 28 is attached. The cooling coil preferably extends through platform 28 at portions 34 and 36 (Figure 2) and may be imbedded therein. The cooling coil further includes a u-bend section 38 and a pair of end sections 40 and 42. End section 40 is connected to a thermal control  
10 bath 44 and end section 42 is connected to coolant receiving tank 46. Flexible hoses 47 and 48 may be provided at the end sections 40 and 42, if desired. The thermal control bath supplies water-glycol (23 C) to the platform to cool the test apparatus during testing.

15 Platform 28 is typically a small copper block which is approximately 4.4 centimeters long (along the axis of the cooling coil), 1.6 centimeters in depth and 3.8 centimeters in width, to minimize its effect on the plasma environment. Platform 28 serves as a base to support a  
20 thermoelectric module 50 including heating elements 52 and assists the module in maintaining a low test temperature for the test sample 10. Module 50 is available commercially as Melcor Model CP10127-05L, and has a surface area of approximately 9 cm and thermally coupled to  
25 platform 28 by standard thermal transfer paste.

The material test specimen 10 is placed upon the thermoelectric module 50 and held in place by the cover glass plate 32 having a specimen surface exposure area. The temperature of the sample is monitored by a  
30 thermocouple probe 54 which is spring loaded against a groove in the thermoelectric module 50 and presses against the sample. The glass cover plate 32 holds the test material 52 firmly against the probe 54. The probe 54 signal wire 56 is insulated and extends back through the  
35 closure member to a meter 58. The cooling coil 26 and platform 28 is located adjacent a vacuum vent 60 to preclude the build up of a stagnant gas layer.

In operation, the sample 10 is placed on the module 50 and the test apparatus is inserted into the reaction chamber with the disc closure member sealing the chamber. The reaction chamber pressure is reduced and then  
5 backfilled by gaseous oxygen of 99.9 percent purity to a stable pressure in the range of 600 to 900 millitorr. The sample 10 is heated or cooled by the thermoelectric device (assisted by the cooled platform 28) and allowed to stabilize at a set temperature for approximately fifteen  
10 minutes. After the system pressure and the material temperature have stabilized, the power is applied to produced the oxygen plasma. Visible glow of oxygen is then observed. Any changes in bulk sample temperature detected by the thermocouple probe are quickly adjusted to maintain  
15 the initial bulk material temperature by the thermoelectric module 50. The base temperature of the sample 10 has been found to be essentially at the temperature of its surface, that is within 3 degrees of the base temperature.

In previous tests, material specimens were exposed  
20 nominally for thirty minutes without interruptions at a specified temperature with accumulation of two to three data points. The PLASMOD is operated at a low wattage so that the ambient plasma temperature is near the lowest sample test temperature. This allows for more accurate  
25 data and closer thermal control of the test sample. Nominal test temperatures were 10, 45, 75 degrees Celcius. No lower temperature was chosen as a test temperature because of the concern for possible gaseous molecular condensation on the material sample. The upper temperature  
30 bound was chosen because the thermoelectric module 50 could become inefficient after lengthy operation at 80 to 85 degrees centigrade and the upper use temperature of plasma exposure was approached. Plasma exposures up to thirty minutes were necessary for such materials as  
35 polytetrafluoroethylene because of its low mass loss rate. Residual gas analyses may be made of the gases in the reaction chamber to provide additional insight into the

fracture of the polymer under atomic oxygen attack if desired.

## ABSTRACT OF THE DISCLOSURE

Apparatus and method for determining the effects of exposure of oxygen plasma on a specimen such as a thin film polymer or thin metals. Apparatus includes a chamber  
5 having a holder supporting the polymer specimen in a plasma environment provided in the chamber. The chamber is regulated to a predetermined pressure and set temperature prior to the introduction of oxygen plasma therein. The specimen is then subjected to the plasma environment for a  
10 predetermined time during which time the temperature of the specimen is sensed and regulated to be maintained at the set temperature. Temperature sensing is accomplished by a probe which senses any changes in bulk sample temperature. Temperature regulation is provided by a thermoelectric  
15 module and by a coolant flow tube.



TECHNICAL ABSTRACT

NASA Case MFS-28368-1

An apparatus and method for determining the effects of exposure of oxygen plasma on a thin film polymer whose bulk is maintained at a predetermined temperature.

The apparatus includes a chamber 8 having a specimen 10 therein. A plasma environment is provided in the chamber 8. A closure member 18 is provided for sealing the chamber after the specimen is introduced into the chamber 108. The closure member also serves as a support for the test apparatus 24 which includes a cooling coil 26. A platform 28 having the test specimen thereon is supported on the cooling coil to be cooled by coolant flowing through the cooling coils. A thermoelectric module 50 is supported 15 on platform 28 to assist in maintaining a low test temperature for the test sample 10. The temperature of the sample is monitored by a thermocouple probe 54 which is in contact with the sample. Any changes in bulk sample temperature caused by the thermocouple probe is quickly 20 adjusted to maintain the initial bulk temperature by the thermoelectric module 50.

The novelty and advantages of the invention lies in the concept of a sample holder for use in a plasma chamber, the sample holder having a cooling coil in combination with 25 a thermoelectric heater and the thermocouple probe.

Inventor: Ann F. Whitaker

Employer: NASA/MSFC

Date Filed: July 28, 1989

Serial Number: 07/386,174

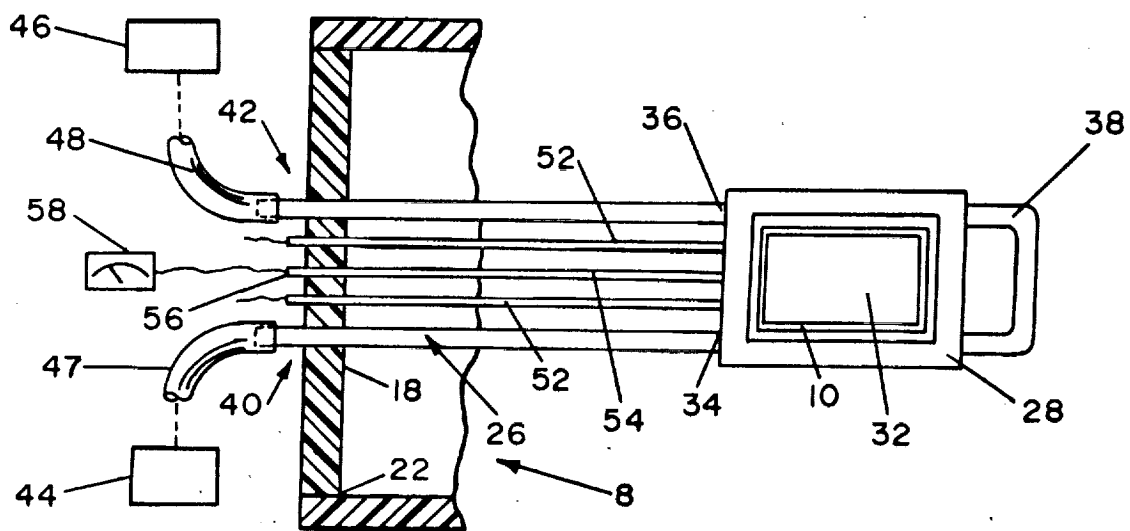


FIG. 2

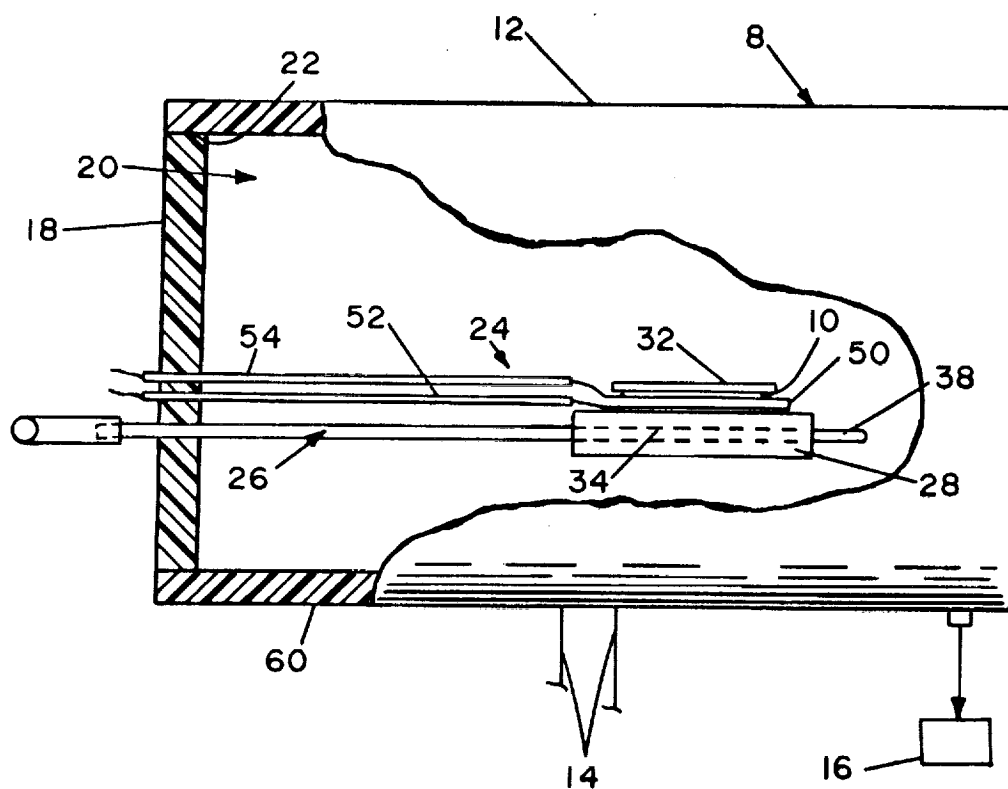


FIG. 1